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Date: 10-19-05

By Sharon Nance

**IN THE UNITED STATES PATENT AND
TRADEMARK OFFICE**

Applicant(s):	Terje A. Skotheim, et al.	Docket No.:	57519.00057 (MT0026.3)
Application No.:	09/864,890	Group Art Unit:	1746
Filing Date:	May 23, 2001	Examiner:	Wills, Monique M.
Title:	LITHIUM ANODES FOR ELECTROCHEMICAL CELLS		

**Declaration under 37 C.F.R.
§1.132 Related to Results of Invention**

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Commissioner:

I, John Affinito, declare as follows:

1. I reside at 5745 North Campbell Avenue, Tucson, Arizona, 85718, am one of the inventors listed in this application and have personal knowledge of the subject matter herein.
2. Evidence is herewith provided that the lithium anode of Applicants' invention is patentably distinct over a lithium anode having no CO₂ treatment and over a lithium anode having CO₂ treatment applied after the lithium is deposited.
3. The evidence herein shows the superior results of utilizing the present invention, which includes the step of co-depositing gaseous material and lithium onto a substrate to form an anode.

4. Examples A-C, below, disclose respectively:
- (i) the manufacture of an anode substrate with a deposited lithium layer and no CO₂ treatment (Example A),
 - (ii) an anode substrate co-deposited with lithium and CO₂ (Example B), and
 - (iii) an anode substrate with a deposited lithium layer post-treated with CO₂ i.e., rather than co-deposited as claimed herein (Example C).

The anode of Example A is a control. The anode of Example B is an example of the co-deposition of a lithium anode as taught in this application. The anode of Example C is one in which CO₂ is used to post-treat the surface of a deposited lithium anode.

As shown in the Tables following the Examples, the co-deposited lithium/CO₂ anode provided superior, unexpected results. Thus Applicants' method as claimed is not obvious in view of U.S. Patent No. 5,824,434 to *Kawakami et al.*

5. Example A: Deposit of Lithium to Form an Anode with no CO₂ Treatment

A vacuum web coating system located in a dry room, having an unwind drive, a liquid cooled drum at -20 °C, load cell rollers for controlling tension, a rewind drive, and two deposition zones, was loaded with an anode substrate of 23 µm PET metallized on one side with 60 nm of inconel and of 15 cm width. The chamber was evacuated to 10⁻⁶ Torr. Lithium was deposited onto the substrate by first heating a thermal evaporation lithium source to 535 °C to allow significant evaporation, and then starting the web drive at 0.5 feet per minute. The lithium evaporation was allowed to stabilize to give a 10 µm coating of lithium on the inconel of the substrate layer. There was no CO₂ treatment.

6. Example B: Co-deposit of Lithium and CO₂ to Form an Anode

A vacuum web coating system located in a dry room, having an unwind drive, a liquid cooled drum at -20 °C, load cell rollers for controlling tension, a rewind drive, and two deposition zones, was loaded with an anode substrate of 23 µm PET metallized on one side with 60 nm of inconel and of 15 cm width. The chamber was evacuated to 10⁻⁶ Torr. Lithium was

deposited onto the substrate by first heating a thermal evaporation lithium source to 535 °C to allow significant evaporation, and then starting the web drive at 0.5 feet per minute. The lithium evaporation was allowed to stabilize to give a 10 µm coating of lithium on the inconel of the substrate layer. Immediately adjacent to the lithium source CO₂ was introduced through a mass flow controller at a flow between 10 and 100 sccm raising the pressure to 0.1 to 50 mTorr. Dark discoloration was immediately seen in the co-deposited lithium with CO₂ from this in-situ deposition process.

7. Example C: Surface of Deposited Lithium Anode Post-Treated with CO₂

A vacuum web coating system located in a dry room, having an unwind drive, a liquid cooled drum at -20 °C, load cell rollers for controlling tension, a rewind drive, and two deposition zones, was loaded with an anode substrate of 23 µm PET metallized on one side with 60 nm of inconel and of 15 cm width. The chamber was evacuated to 10⁻⁶ Torr. Lithium was deposited onto the substrate by first heating a thermal evaporation lithium source to 535 °C to allow significant evaporation, and then starting the web drive at 0.5 feet per minute. The lithium evaporation was allowed to stabilize to give a 10 µm coating of lithium on the inconel of the substrate layer. Upon completion of the deposition, the lithium coated substrate (lithium anode) was re-wound on the unwind drive while the vacuum was maintained in the apparatus. With the lithium source off, the lithium coated substrate (anode) was subjected to RF magnetron plasma treatment with the CO₂ gas. Forward RF power was between 50 and 100 W at a pressure of 0.1 to 50 mTorr. The post-treated lithium had a dark appearance.

8. Fabrication of Electrochemical Cells from the Anodes of Examples A, B, and C

Small flat cells were assembled as follows: A composite cathode was prepared by coating a 3.68 cm wide cathode active layer on a 4.19 cm wide Al/PET substrate. A cathode slurry was prepared from 70 parts by weight of elemental sulfur, 15 parts by weight of conductive carbon, 10 parts by weight of graphite, 4 parts by weight of TA22-8 resin, and 1 part by weight of Ionac PFAZ-322. The solids content of the slurry was 14% by weight in a solvent mixture of 80% isopropanol, 12% water, 5% 1-methoxy-2-propanol and 3% dimethylethanolamine (on a weight basis). The slurry was coated by a slot die coater onto both sides of the substrate. The coating was dried in the ovens of the slot die coater. The resulting

dry cathode active layer had a thickness of about 15 microns on each side of the current collector, with a loading of electroactive cathode material of about 0.89 mg/cm². 4.5 cm lengths of this composite cathode were used in building the electrochemical cells.

Lithium anodes of 10 cm in length and 4.19 cm in width were cut from the anode materials of Examples A, B, and C, respectively. Small flat cells were assembled by folding each respective anode around a cathode with a 10 µm polyolefin separator inserted between the anode and cathode. The cells were placed into a polymer-coated aluminum foil bag. 0.4mL of a 1.4 M solution of lithium bis(trifluoromethylsulfonyl)imide, in a 42:58 volume ratio mixture of 1,3-dioxolane and dimethoxyethane, was added as electrolyte and each electrochemical cell was vacuum sealed. Therefore, each electrochemical cell was the same except for the anode.

Testing was performed at a discharge current of 0.42 mA/cm² to a voltage of 1.5V and charged at a current of 0.24 mA/cm² to 110% last half cycle capacity.

9. Performance of Electrochemical Cells made from the Anodes of Examples A, B and C

The specific discharge capacity versus cycles of the cells fabricated from anodes of Example A, Example B, and Example C are summarized in Table 1. These results show that electrochemical cells utilizing the anodes with co-deposited lithium (Example B) according to the invention outperformed cells with untreated lithium anodes (Example A) and cells with lithium anodes post-treated with CO₂ (Example C):

TABLE 1			
SPECIFIC CAPACITY			
Cycle No.	Li Anode (A)	Li Co-deposit in situ (B)	Li with post plasma CO ₂ (C)
10	485 mAh/g	528 mAh/g	470 mAh/g
20	430 mAh/g	444 mAh/g	383 mAh/g
30	226 mAh/g	357 mAh/g	174 mAh/g
40	96 mAh/g	170 mAh/g	87 mAh/g

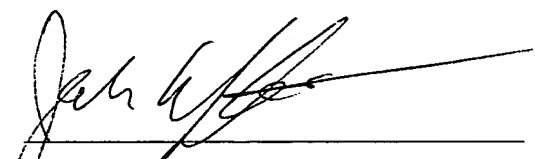
In Table 2 the ratios of the discharge capacity of electrochemical cells utilizing untreated lithium anodes (Example A) are compared with electrochemical cells utilizing co-deposited

lithium anodes (Example B) and lithium anodes post-treated with CO₂ (Example C). These comparisons show that the anode of Example C (with post-treatment of CO₂) provides no improvement in performance over a lithium anode with no CO₂ treatment (Example A), whereas the co-deposition of lithium and CO₂ (Example B) enhances performance of the electro-chemical cell:

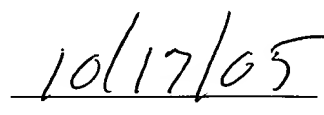
TABLE 2		
RATIO OF CAPACITY TO LI ANODE (A)		
Cycle No.	Co-Deposited (B)	Post Treatment (C)
10	1.09	0.97
20	1.03	0.89
30	1.57	0.77
40	1.77	0.91

In summary, the present invention yields superior and unexpected results over an anode with deposited lithium and no CO₂ treatment and over an anode with lithium deposited first and then being treated with CO₂.

I declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed true; and that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, as provided by 35 U.S.C. 25 and 18 U.S.C. § 1001, and that such willful false statement may jeopardize the validity of the application or any patent issuing thereon.



John Affinito



Date